GEORGIA INSTITUTE OF TECHNOLOGY
ENGINEERING EXPERIMENT STATION

PROJECT INITIATION

Date: May 9, 1975

Project Title: Characterization of Samples Containing Noble Metal Values

Project No.: A-1731

Project Director: Mr. M. F. Munoz

Sponsor: Wheat-Junkin

Agreement Period: From April 23, 1975 Until May 28, 1975

Type Agreement: Standard Industrial Research Agreement

Amount: $7,675

Reports Required: Letter Progress Report; Final Report

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RA-3 (3-75)

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SPONSORED PROJECT TERMINATION

Date: July 6, 1977

Project Title: Characterization of Samples Containing Noble Metal Values

Project No: A-1731

Project Director: Mr. M. F. Munoz

Sponsor: Wheat-Junkin

Effective Termination Date: 6/30/77

Clearance of Accounting Charges: All clear.

Grant/Contract Closeout Actions Remaining:

_ Final Invoice and Closing Documents
_ Final Fiscal Report
_ Final Report of Inventions
_ Govt. Property Inventory & Related Certificate
_ Classified Material Certificate
X Other Refund Advance Payment (Ms. Miley)

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CA–4 (3/76)

A-1731

ANALYSIS OF SAMPLES

CONTAINING NOBLE METAL VALUES

Prepared by

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Final Report

Phase I

Project A-1731

June, 1975

I. Introduction and Specific Obejctives

Gold, silver, and metals of the platinum group occur in the ore present in the mineral property of the sponsors. D. C. arc spectrographic analysis of surface samples have shown the presence of the above mentioned metals in the ore in concentrations sufficient to warrant more detailed exploration of the mineral property.

The sponsors requested that a proposal be written by EES encompassing the necessary work to determine:

1. The amount and extent of the noble metals in the ore;
2. Processing methods to extract the metal values from the ore.

A proposal which covered only the characterization of core samples (based on analyses of five cores, each 100 feet long) was submitted to the sponsors on February 11, 1975. The proposal included a detailed characterization of the ore, which would have allowed the design of a process to extract the metal values.

On April 23, 1975, the sponsors decided to fund analysis work on one core obtained by them, and drilled on their property. Project A-1731 was initiated, having as its purpose and objective to determine the content of the noble metals (gold, silver, and the metals of the platinum group) present in one core sample supplied by the sponsor, so that the extent of the concentration of these metals over a given depth could be determined. The results presented herein will serve as basis for further depth analyses of the mineral property and as basis for processing methods for the ore.

II. Experimental Procedure

The procedure followed in the analyses of the sample consisted in visual description of the core, sampling, and analyses of the samples obtained.

A. Visual Description. The core sample was obtained by a geologist retained by the sponsor, and the cores brought to the Engineering Experiment Station laboratory by the sponsors. Care was taken to wrap the samples in polyethylene plastic, to preserve the soil moisture. The core samples were unwrapped, cut lengthwise and described. Results of the descriptions are shown in Table I. One half of the core was combined into five foot composites for analyses and placed into a dryer at 105°C. The other half was re-packed in plastic and returned to the sponsor.

B. Sampling. The moisture was determined and the five foot composites dried at 105°C. The dried composites were ground to pass a 100 mesh screen, and split into four subsamples each. One set of subsamples was used for D. C. arc spectrographic analysis, one set for Atomic Absorption and two sets were sent to the sponsor, at his request. The sampling procedure is shown in Figure 1.

C. Analyses. The dried, ground samples were analyzed by D. C. arc spectrography using standards of silver, gold, platinum, rhodium, ruthenium, palladium, osmium and iridium. The results obtained are shown in Table II.

A second set of dry, ground samples were analyzed by Atomic Absorption methods. The procedure followed included fusion with lithium metaborate in a graphite crucible. This procedure is described in Appendix 1.

Results obtained using this method made it necessary to change the mode of analysis by digestion of the fused sample in aqua regia for five days prior to atomic absorption analyses. The final analysis procedure is described in Appendix 2.

FIGURE I

FLOW PROCESS

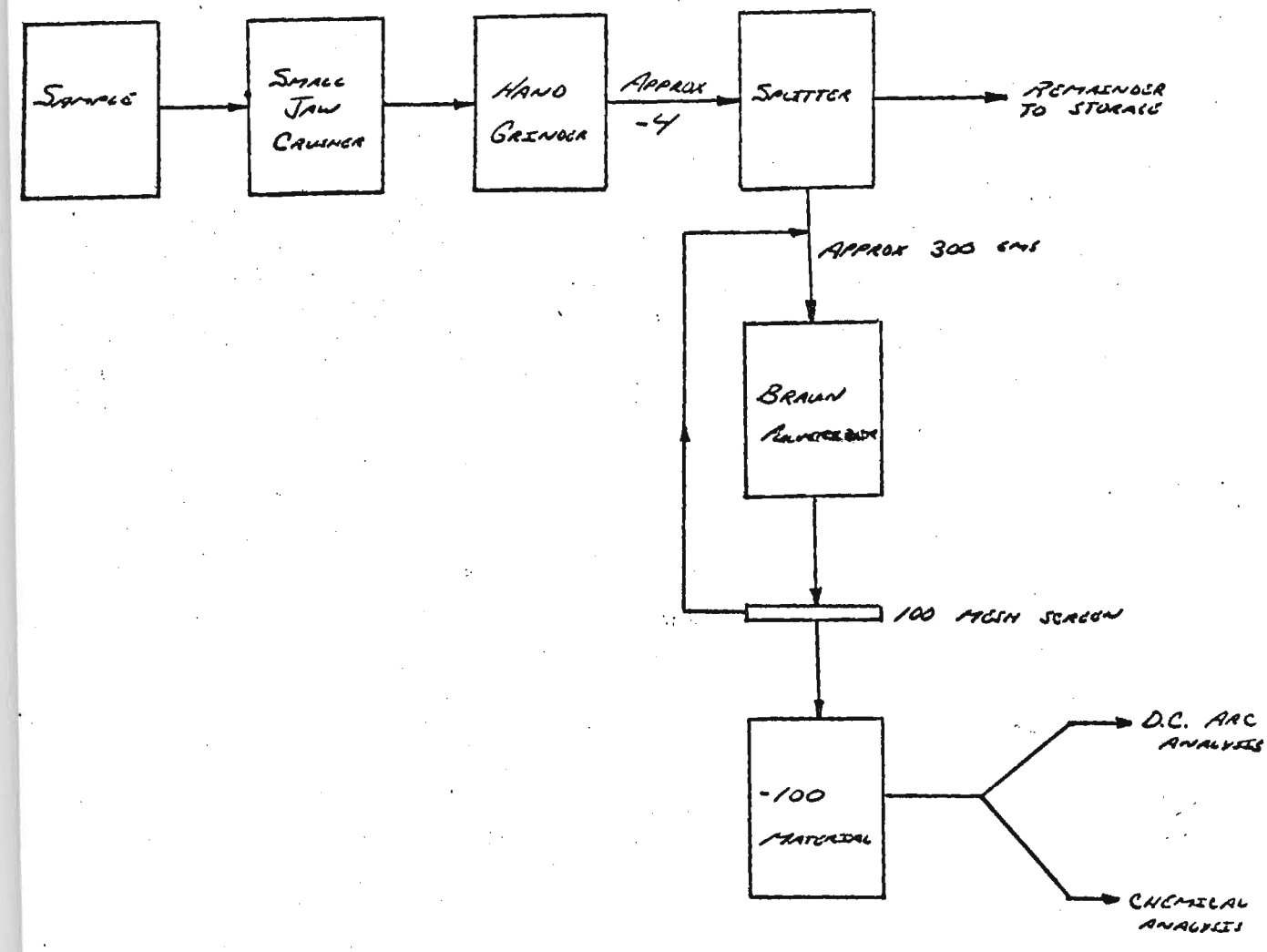

TABLE I

VISUAL DESCRIPTION OF CORE SAMPLE

- Well No. 2 -

Average Percent Moisture: 15.0

Core Interval Feet and Inches	Core Recovery (inches)	Description
0' - 5'	65	Clay - Light chocolate brown to yellow.
5' - 8'		Sandy clay - yellowish - brown to gray.
9' - 20'	-	Wash sample - yellowish sand.
24'	-	Wash sample - yellowish sand.
27'	-	Wash sample - yellowish sand.
31'4" - 33'4"	9	Hard sandy clay - gray.
32' - 35'	-	Wash sample - dark gray.
33'4" - 38'4"	9	Hard sandy clay - gray.
38'4" - 43'4"		Hard sandy clay - gray.
43'4" - 47'	73	Gray sandy clay.
47' - 52'	22	Gray sandy clay - It appears that the amount of clay increases as depth approaches 52'. Clay nodules dark gray and black.
52' - 62'	3	Very little core - light bluish gray with white.
52'	-	Wash sample - large amount of gravel.
62' - 67'	42	Reddish brown to dark brown clay.
67' - 72'		Choc. brown clay with flecks of gray clay.
72' - 77'	40	Choc. brown clay interspersed with gray clay.
77' - 82'		Choc. brown clay interspersed with gray clay.

TABLE I (Continued)

Core Interval Feet and Inches	Core Recovery (inches)	Description
82' - 87'	35	Light brown - gray purple clay with yellow stains increasing as depth increases.
87' - 92'	80	Choc. brown - some purple clay with white spots.
92' - 97'	57	Dark choc. brown clay with some gray clay. Purple clay increases up to 96' then dissapears.
97' - 102'	55	Dark choc. brown clay with some gray streaks.
102' - 107'	55	Choc. brown clay with yellow and purple flecks.
107' - 112'	46	Brown to light brown clay with some yellow flecks - some small pebbles.
112' - 117'	50	Light brown to yellow clay.
117' - 122'	33	Hard dark gray to light gray clay with yellowish flecks.
122' - 127'	39	Hard dark gray clay - homogeneous in color.
127' - 132'	16	Gray to light gray clay - some yellowish flecks present.
132' - 137'	29	Dark gray hard clay some limestone present.
137' - 139'6"	10	Light to medium gray clay with limestone. Limestone heavily fossilized.

Table III shows the analysis results obtained presented as Troy ounces of metal per short ton of ore.

Selected samples were also analyzed through x-ray diffraction, to determine mineral content. Table IV shows the results obtained.

III. Discussion of Results

The results obtained indicate that the samples submitted by the sponsors and analyzed at the EES laboratories contain substantial amounts of silver gold, platinum, rhodium, ruthenium, palladium, osmium and iridium at the depth intervals identified by the sponsors.

The recovery of core samples submitted correlated well with a copy of the driller's log, which was submitted by the sponsors, with the exception of core interval 33'4" to 43'4". The core recovery reported by the driller is 2'4" while the measured amount of core at EES was nine inches. The driller's log showed greenish gray-brown sand at 102' - 107' red-green and blue clay at 107' - 112', and waxy-green clay at 112' - 117'. Visual observations on the core at the Mineral Beneficiation Laboratory indicated chocolate brown clay at 102' - 107', brown to light brown clay at 107' - 112', and a light brown to yellow, homogeneous clay at 112 - 117'.

The color discrepancies may be caused by oxidation of some of the clays core samples in transit. Tables II and III show results of analysis, semi-quantitative D.C. arc spectrographic in Table II and atomic absorption in Table III.

RESULTS OF D. C. ARc SPECTROGRAPHIC (SEMI-QUANTITATIVE) ANALYSES OF CORE SAMPLE

RESULTS EXPRESSED IN TROY OUNCES OF METAL PER TON OF ORE

NOTE: These results are approximate.

Core Interval (In Feet & Inches)	Silver (Ag)	Gold (Au)	Rhodium (Rh)	Palladium (Pd)	Osmium (Os)	Ruthenium (Ru)	Platinum (Pt)	Iridium (Ir)
0' - 5'								
5' - 8'		0.3-2.9						
9' - 20'(wash sample)		0.3-2.9					0.1-0.3*	
24' (wash sample)								
27' (wash sample)	0.1-1.4							
31'4" - 33'4"	0.1-1.4				0.03-0.3		0.3-1.5	
32' - 35'(wash sample)								
33'4" - 38'4"								1.5-5.8
38'4" - 43'4"	0.1-1.4	0.3-1.4			0.03-0.3			0.1-1.5
43'4" - 47'	0.1-1.4				0.1-1.4		1.5-2.9	
47' - 52'	1.4-14	0.1-1.4					1.5-8.7	
52' - 62'	0.1-1.4	1.4-8.7			0.03-0.3		0.1-1.5	
52'(wash sample)	0.1-1.4						0.1-1.5	
62' - 67'	8.7-29				0.1-1.4			
67' - 72'	8.7-29							
72' - 77'						0.3-1.4		
77' - 82'		0.1-1.4	1.4-14		0.1-1.4		0.1-0.3*	
82' - 87'	1.4-14				0.1-1.4		0.1-1.5	1.5-5.8
87' - 92'		0.3-2.9			1.4-5.8		0.1-1.5	0.1-1.5
92' - 97'					0.1-1.4			0.1-1.5
97' - 102'	1.4-14	0.1-0.6*					0.1-1.5	
102' - 107'	1.4-14	1.4-8.7			0.1-1.4		0.1-8.7	
107' 112'	1.4-14		0.3-2.9	0.3-1.5	0.1-1.4	0.03-0.3		
112' - 117'	0.1-1.4	0.3-2.9						
117' - 122'	8.7-29							
122' - 127'			0.3-1.5					
127' - 132'	0.1-1.4			0.3-1.5				
132' - 137'							0.1-5.8	
137' - 139'6"		1.5-5.8					0.1-5.8	

*Possible, but right at detection limit.

TABLE III

RESULTS OF ATOMIC ABSORPTION ANALYSES OF CORE SAMPLES

Results are shown in Troy ounces of metal per short ton of dry ore.

Core Interval, Feet and inches	Silver (Ag)	Gold (Au)	Rhodium (Rh)	Palladium (Pd)	Osmium (Os)	Ruthenium (Ru)	Platinum (Pt)	Iridium (Ir)
0' - 5'	-	-	-	-	-	-	-	-
5' - 8'	-	3.6	-	-	-	-		-
9' - 20' wash	-	7.4	-	-	-	-	0.7	-
24' wash	-	-	-	-	-	-	-	-
27' wash	0.9	-	-	-	-	-	-	-
31'4" - 33'4"	4.96	-	-	-	0.18	-	1.8	-
32' - 35' wash	-	-	-	-	-	-	-	-
33'4" - 38'4"	-	-	-	-	-	-	-	5.8*
38'4" - 43'4"	0.5	3.2	-	-	0.7	-	-	5.8*
43'4" - 47'	1.0	-	-	-	1.3	-	1.9	-
47' - 52'	0.7	3.9	-	-	-	-	1.3	-
52' - 62'	1.9	4.1	-	-	2.18	-	1.5	-
52' wash	1.6						2.5	-
62' - 67'	3.8	-	-	-	1.02	-	-	-
67' - 72'	1.02							
72' - 77'	-	-	1.45	-	-	0.43	-	-
77' - 82'	0.72	7.29	2.91	-	0.5	-	2.18	-
82' - 87'	1.75	-	2.18	1.31	1.31	-	3.35	7.29*
87' - 92'	0.88	4.81	2.18	-	0.5	-	0.87	7.29*
92' - 97'	-	-	-	-	1.97	-	-	7.29*
97' - 102'	1.17	2.92	1.81	-	-	-	4.46	-
102' - 107'	7.87	6.12	3.26	-	1.24	-	1.89	-
107' - 112'	0.58	-	-	1.07	0.66	1.46	-	-
112' - 117'	4.67	5.10	-	-	-	-	-	-
117' -122'	0.23	-	-	-	-	-	-	-
122' - 127'	-	-	1.45	1.07	-	-	-	-
127' - 132'	0.29	-	-	1.66	-	-	-	-
132' - 137'	-	-	-	-	-	-	3.44	-
137' - 139'6"	-	1.74	-	-	-	-	2.82	-

*Possible, but right at detection limit.

TABLE IV

MINERAL ANALYSIS OF SELECTED SAMPLES OF CORE
BY X-RAY DIFFRACTION

Core Interval, Feet	Minerals Detected
62' - 67'	Ag_2S (silver sulfide)
67' - 72'	Ag_2S (silver sulfide)

It should be pointed out that the apparent disparity between the values of a given metal analysis as shown in Table II and as shown in Table III is due to the semi-quantitative nature of the D. C. arc analysis, which only points out the presence and range of concentration of a given metal in the sample being analyzed. D. C. arc spectrographic analysis can be made quantitative, but that requires a long and tedious procedure to match the impurity matrix with the standard of the elements sought.

Atomic absorption, analysis, on the other hand, is easily made quantitative with a great degree of accuracy and precision. This method requires, however, that all the sample be in solution, i.e., the sample be "opened" correctly.

Fusion with lithium metaborate in a graphite crucible has been used quite extensively as a method to "open" silicate samples. Consequently, this method was used in the samples submitted. The results obtained indicated very low concentrations of gold that did not correspond to the D. C. arc data. This was interpreted as a failure to place the sample in solution. In other words the lithium metaborate fusion did not dissolve the refractory metal compounds. Through experimentation it was found that if the melt of the lithium metaborate fusion was digested five days in aqua regia (a mixture of HCl and HNO_3), filtered, and the filter ashed and fused with lithium metaborate and added to the solution, the concentration of the metals increased to levels comparable to the values reported for D. C. arc results.

Therefore, the values obtained in the single fusion of the sample with lithium metaborate were discarded. Another test was performed on the samples by digestion with aqua regia for one week (without fusing the sample first) but the results were low and disregarded.

The values reported in Table III are the results of the fusion of the sample with lithium metaborate, as shown in detail in the appendix.

As an additional proof of the presence of the silver as an independent mineral in the samples, Table IV shows that silver sulphide was identified in core intervals 62' to 67' and 67' to 72' by x-ray diffraction.

The opening and solubilizing of the noble metals in these samples were difficult, and it is estimated that if there is variation from location to location in the mineral property, more research will have to be done to determine methods of analyses that will assure that all the elements sought are in solution.

It should be mentioned that due to the detection limit of the atomic absorption unit for the element iridium (1.0 part per million in solution) the value reported for iridium is not considered reliable. In order to improve the reliability of the result, the metals in the sample should have been concentrated, so that the amount if iridium would have been above the detection limit in solution. The time allotted for the work was not, however, enough to attempt a concentration operation.

The analysis of silver in the samples treated with aqua regia required special processing. The silver was precipitated quantitatively and re-dissolved as the complex ion to be sure that it was in solution. Details of the procedure are shown in Appendix 2.

IV. Conclusions

1. The results presented in Table III show conclusively that the core samples submitted contain substantial amounts of silver, gold, platinum, palladium, osmium, rhodium, ruthenium, and possibly, iridium.

2. The solution of the ore sample ("opening") is a critical step in the analysis procedure, since the elements analyzed are extremely resistant to chemical attack.

3. A lithium metaborate fusion - aqua regia digestion - lithium metaborate fusion of residue method appears to "open" adequately the sample.

V. Recommendations

1. On the basis of the results presented herein, it is recommended that the work proposed under the February 11, 1975, proposal should be undertaken as Phase II and five cores should be characterized in the form described in the body of the proposal.

2. Additional work should be continued since the nature of the noble metal compounds may change throughout the deposit. This work is in addition to the work described above in recommendation No. 1 and is not included in the cost estimate referred to above.

Appendices

APPENDIX I

ATOMIC ABSORPTION ANALYSIS

(Single Lithium Metaborate Fusion Procedure)

A 0.1000 gram sample of ground, dry, ore was weighed into a Pyrex evaporating dish. Then 0.50 grams of lithium metaborate were weighed into the dish. The ore was mixed with the lithium metaborate with a monel spatula until a uniform color was obtained.

After the sample had been thoroughly blended the contents of the evaporating dish were quantitatively transferred to a graphite crucible. The sample, ready for fusion was kept in a dessicator until it was fused.

The sample was fused for 30 minutes at 950°C. The graphite crucible was removed from the furnace and the fused bead formed by the ore and the lithium metaborate allowed to cool briefly before pouring into 50 ml. of four percent nitric acid contained in a plastic beaker. The contents of the beaker were stirred until the melt dissolved completely in the solution. The contents of the beaker were then transferred quantitatively to a 100 ml. volumetric flask. The beaker was rinsed with 4 percent nitric acid and the washings were added to the volumetric flask. Distilled water was added to the volumetric flask up to volume, and the flask shaken. The contents of the volumetric flask were filtered through Whatman filter paper (No. 40 Ashless) to remove any contaminant graphite particles from the crucible. The filtrate was collected into a cleaned and rinsed four ounce plastic bottle provided with a cap to avoid contamination. The atomic absorption determinations were performed on the solution contained in the four ounce bottle.

APPENDIX II

ATOMIC ABSORPTION ANALYSIS

(Lithium Metaborate Fusion - Aqua Regia Digestion - Lithium Metaborate Fusion Procedure)

1. Sample Preparation

A 0.2500 (± 0.0001) gram sample of ground, dry ore was weighed into an evaporating dish. To the sample 1.0000 gram of lithium metaborate (anhydrous) was added and thoroughly mixed with a monel spatula. The mixture was quantitatively transferred while still red hot to 50 ml. of four percent nitric acid and dissolved with constant stirring. The solution was almost taken to dryness and 50 ml. of aqua regia (four parts HCl to one part HNO_3) was added to the beaker. This volume of aqua regia was maintained constant through a digestion period of five days. The sample was maintained at a constant 60°C temperature in a water bath. At the end of the digestion, concentrated nitric acid was added to a volume of 80 ml.; the sample was digested in concentrated nitric acid overnight. In the morning, the sample was evaporated almost to dryness on a hot plate and 50 ml. of distilled water added to dissolve the remaining residue on the walls and bottom of the beaker. The solution was gently heated and stirred to obtain optimum dissolution of the residue. The final volume of the sample solution was approximately 100 ml. The solution was then filtered through Whatman #40 filter paper (Ashless).

The filtrate (about 140 ml.) was evaporated to a volume of about 50 ml.

The filter paper was placed in an individual porcelain crucible and ashed at 520°C for two hours.

The residue from the crucible was then mixed with 0.5 grams of lithium metaborate (anhydrous) in a graphite crucible and fused at 950°C for 30 minutes. The red hot fused bead was transferred into the filtrate obtained earlier. The melt was dissolved in the filtrate with constant stirring. The solution was then transferred to a 100 ml. volumetric flask and filuted to volume. Since silver is precipitated by the chloride ion (present in aqua regia), two sets of samples were prepared from the 100 ml. solution to be analyzed by atomic absorption:

a. A twenty five ml. aliquote of the solution was pipetted into a small (150 ml.) plastic beaker. Five ml. of concentrated HCl were added with stirring. A small white precipitate was noted in the sample (except the blakn, which was clear). Approximately 12 ml. of freshly filtered NH_4OH were added with stirring and the solution cleared, placing all the silver present as the complex ion $[Ag\ (NH_3)_2]+$. The solution was then filtered through a glass fiber filter into a 100 ml. volumetric flask. The filtrate was diluted to mark and transferred to a four ounce plastic bottle and analyzed by atomic absorption for silver content.

b. The remainder of the final solutions were filtered through glass fiber filters and collected in plastic four ounce bottles for atomic absorption analysis of gold, platinum, osmium, rhodium, palladium, ruthenium and iridium. Note: A lanthanum oxide (La_2O_3) buffer was added to samples to be analyzed for iridium, platinum, ruthenium, palladium, and rhodium prior to the analysis.